LAWRENCE
LIVERMORE
NATIONAL
LABORATORY

LLNL-PROC-413618

Static Material Strength Determined Using a DAC

H. Cynn, W. Evans, J. P. Klepeis, M. Lipp, P. Liermann, W. Yang

June 5, 2009

US-Russia Material Sciences Conference
Prague, Czech Republic
August 30, 2009 through September 4, 2009

Disclaimer

Static material strength determined using a DAC

Hyunchae Cynn[1], William Evans[1]JHP Klepeis[1], Magnus Lipp[1],
Peter Liermann[2*], Wenge Yang[2]
[1]Lawrence Livermore National Laboratory, Livermore, CA 94550, USA
[2]HPCAT, Bld. 434E, Advanced Photon Source Argonne National Laboratory,
Argonne, IL 60439-4803, USA
*Currently at DESY, HASYLAB, Petra III, P02 Notkestr. 85, Bldg. 47c, Rm. L115
22607 Hamburg, Germany

Abstract

By measuring sample thickness and pressure gradient using x-ray absorption and x-ray diffraction, respectively, the accurate static yield strengths of Ta and Fe were determined at high pressure. This improved method has several advantages over other similar methods to quantitatively determine static material strength.

Introduction

There have been continuous efforts to understand material strength at high pressure under static and dynamic conditions. Most static strength measurements are based on x-ray diffraction data, which easily connects easy to high pressure measurements using a diamond anvil cell (DAC). By simplifying the stress conditions of a sample compressed uniaxially in a DAC, the deviatoric stress, σ_3-σ_1 is approximated as yield strength of the sample. [1] Due to the symmetry along the axial compression of the diamond anvil cell (DAC) and the sample, the determined yield strength is represented at a pressure that equals the mean normal stress.

According to Singh and others [2,3,4], the lattice parameter, a(hkl) of the cubic system calculated from the measured d-spacings can be represented by the following relation:

$$a(hkl) = M_o + M_1[3(1-3\sin^2\theta_{hkl})\Gamma(hkl)], \quad (1)$$

where,

$$\Gamma(hkl) = (h^2k^2 + k^2l^2 + l^2h^2)/(h^2 + k^2 + l^2)^2. \quad (2)$$

The intercept, M_o and the slope, M_1 of the equation (1) with knowledge of elastic

compliances, S_{ij} of the sample, and the structure factor, Γ(hkl) in the equation (3) are used to calculate the deviatoric stress represented by the following relation:

$$\sigma_3-\sigma_1 = -3M_1/\alpha M_o(S_{11} – S_{12} – S_{44}/2). \quad (3)$$

The term α is introduced to approximate the stress and strain conditions at the grain boundary. Under the Reuss approximation which approximates iso-stress conditions across the grain boundary, α = 1. If the strain across the grain boundary is approximated same under the Voigt condition, α approaches zero.

Dewale et al. [5] proposed a single crystal strain measurement using x-ray diffraction to estimate material strength with the knowledge of elastic constants of the sample. A laser interference pattern was used to estimate the plastic strain of the sample.

Meade and Jeanloz [6] approximated the difference between the axial and the radial stress under uniaxial compression of a non-hydrostatically compressed sample inside a gasket. In the Tresca approximation, they evaluated the deviatoric stress through the following relation:

$$\sigma_3-\sigma_1 = h(dP/dr) \quad (4)$$

where, h represents the thickness of the sample and dP/dr is the pressure gradient of the sample. Meade and Jeanloz [6] used the ruby fluorescence method to determine the pressure gradient, measured the thickness of the recovered gasket and applied the equation of state to back-calculate the thickness during the compression.

We decided to compare these methods to outline their advantages and weakness and to propose an improved methodology to determine the static material strength at high pressure using a DAC.

Experiments

Polycrystalline Ta and Fe powders (99.99 % purity with a nominal grain size of 1.5μm) were commercially available from Alfa Aesar. The samples were packed in a hole (300 μm in diameter and 10 ~ 40 μm thick) prepared in a Be gasket without a pressure medium. The size of the anvil was 400 μm. Hardened Be gaskets (3mm in

diameter and 1.5 mm thick) were pre-indented to 100 μm and provides the maximum x-ray transmission with a material strength sufficient for achieving high pressures. [7] The volume across the sample was determined via energy-dispersive x-ray diffraction and the pressure was calculated using the equation of state of the sample. [8,9] To measure thickness changes during compression, the x-ray transmission was recorded across the sample. The measured x-ray transmission through the diamond anvils and sample under compression was normalized against the transmission through the x-ray transparent Be gasket plus the anvils. The sample thickness, h was calculated using the following relation:

$$I = I_o \exp(-\mu_l h), \quad (5)$$

where I_o and I, respectively, represent the initial and attenuated x-ray beam intensities. μ_l denotes the linear x-ray absorption coefficient. The mass absorption coefficient, μ_m is equal to μ_l/ρ. ρ is density of the sample.

Results

The x-ray transmission of Ta at 52 GPa is shown in Fig. 1. Using equation (5), the thickness of the Ta sample at 52 GPa was estimated (Fig. 2).

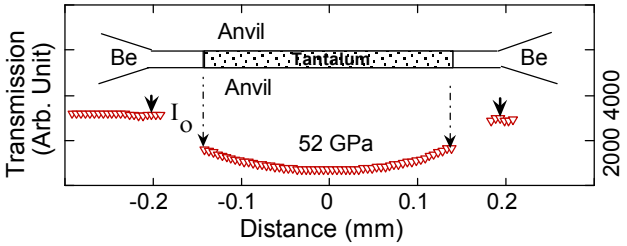

Fig. 1 Transmission of Ta at 52 GPa across the 0.3 mm opening of the Be gasket. Io is the x-ray transmission intensity through the Be gasket and diamond anvils.

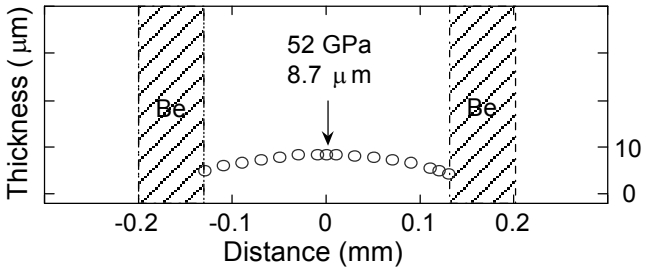

Fig. 2. The thickness of a Ta sample at 52 GPa as obtained by using equ. (5) is plotted as a function of the distance from the center. The size of the open circle represents the uncertainty in thickness determination.

The sample thickness at the center is about 8.7 μm at 52 GPa with a measurable cupping by about 3 μm over 150 μm to the diamond culet edge.

To obtain the pressure gradient of Ta, the lattice strain was measured at every 5 – 20 μm steps using x-ray diffraction along the compression direction. Fig. 3 shows spatially resolved pressure distribution at three pressures whose maximum values at the centers are 52, 27, and 13 GPa.

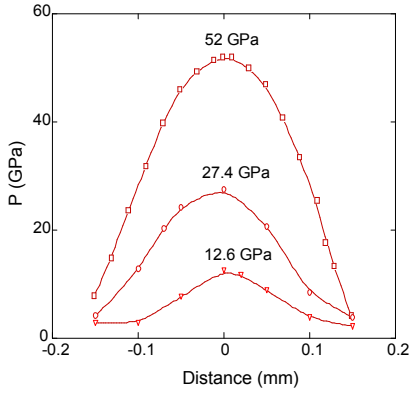

Fig. 3. Spatially resolved pressure distribution with the maximum pressure at the center of the sample. For visual aid solid lines are used to connect the data points.

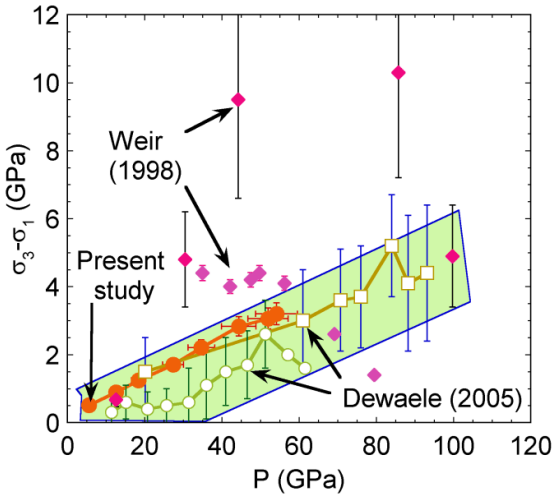

Fig. 4 Deviatoric stress, σ_3-σ_1 of Ta as a function of pressure. Present results are shown as solid orange circles. Two sets of data by Weir et al [10] are shown as red and pink diamonds. Another two sets of data by Dewaele and Loubeyre [5] are shown as open circle and square.

Based on the above mentioned methods to measure sample thickness and pressure gradient, the deviatoric stresses of uniaxially compressed Ta powder were determined. The resulting deviatoric stress of Ta is plotted as a function of pressure in Fig. 4 superimposed with the stresses estimated by Weir et al. [10] and Dewaele and Loubeyre [5]. Linear relation is apparent in present study and in Dewaele and Loubeyre.

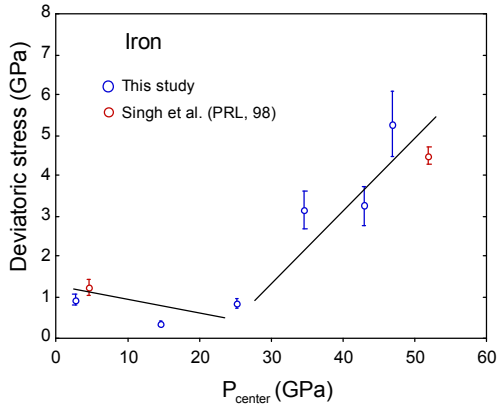

Fig. 5 Deviatoric stress, σ_3-σ_1 of Fe as a function of pressure. Singh et al [11] used equ (3) based on Au elastic compliances.

Deviatoric stress of Fe was also determined using the same methods as in Ta (Fig.4) and compared with the stress determined by Singh et al. [11]. Below 20 GPa, the deviatoric stress of Fe appears decreasing as pressure increases. The stress increases rapidly after 20 GPa up to 50 GPa.

Discussion

High pressure static deviatoric stresses of Ta and Fe were determined using pressure gradient and absorption measurement. The methods employed in the present study have been modified compared to previous methods. [6, 10] The pressure was measured using the EOS of the sample itself unlike the ruby method used by Meade and Jeanloz. [6] One of the disadvantages of the ruby method is the peak broadening at high pressure, especially at non-hydrostatic conditions, which may lower the resolution of the peak position and accordingly result in a less accurate pressure determination. To measure sample thickness, the present study used x-ray absorption. Weir et al. [10] measured the recovered gasket thickness and applied the EOS of the gasket material to estimate the thickness during compression. This can be rather uncertain since plastic deformation and rebound after the recovery are not known. The single crystal strain method by Dewaele and Loubeyre [5] yields the deviatoric stress of Ta in good agreement with the present study. A linear fit to the present data yields a following relation: $\sigma_3 - \sigma_1$ (GPa)= 0.0564*P(GPa) + 0.2006 GPa. The uncertainty in the deviatoric stress increases with increasing pressure because of a less accurate estimation of the sample thickness at high pressure due to thinning of sample and cupping of anvils.

The present modified method seems to yield deviatoric stress results, which agree very well with those determined using different methods. As shown in Fig. 5, a comparison of deviatoric stress of Fe at high pressure suggests that the deviatoric stress determined by Singh et al [11] agree well with the values obtained by the present methods. Recent phonon measurements using near-edge resonant inelastic x-ray scattering of Fe show indeed average velocity decreases from ambient conditions to 6 GPa at 300 K [12], which suggest that the α-Fe instability may be related to the softening of the mechanical properties.

The modified methods to measure sample thickness and pressure gradient are essentially *in-situ* methods unlike others [6,10]. Moreover, this newly established

method does not depend on knowledge of the elastic constants of sample. Last but not least, this new and improved method is easy to adapt at high pressure using a DAC.

References

[1] Ruoff A L 1975 *J. Appl. Phys.* **46** 1389

[2] Singh A K and Kennedy G C 1974 *J. Appl. Phys.* **45** 4686

[3] Singh A K, Balasingh C, Mao H K, Hemley R J and Shu J 1998 *Phys.Rev. Lett.* **80** 2157

[4] Singh A K and Takemura K 2001 *J. Appl. Phys.* **90** 3269

[5] Dewaele A and Loubeyre P 2005 *Phys. Rev.* B **72** 134106

[6] Meade C and Jeanloz R 1988 *J. Geophys. Res.* **93** 3261

[7] Mao H K, Shu J, Shen G, Hemley R J, Li B, Singh A K 1998 *Nature* **396** 741

[8] Cynn H and Yoo C S 1999 *Phys. Rev.* B **59** 8526

[9] Jephcoat A P, Mao H K and Bell P 1986 *J. Geophys. Res.* **91** 4677

[10] Weir S T, Akella J, Ruddle C, Goodwin T and Hsiung L 1998 *Phys. Rev.* B **58** 11258

[11] Singh A, Mao H K, Shu J and Hemley R J 1998 *Phys. Rev. Lett.* **87** 2157

[12] Shen G, Sturhahn W, Alp E E, Zao J, Toellner T S, Prakapenka V B, Meng Y and Mao H K 2004 *Phys. Chem. Minerals* **31** 353

Acknowledgments

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory in part under contract W-7405-Eng-48 and in part under Contract DE-AC52-07NA27344. We also acknowledge support from the LLNL LDRD office (07-ERD-029). The synchrotron radiation experiments were performed at NSLS, SSRL, and HP-CAT.